EVALUATION OF THE MICROTRAC (SPA) FOR PARTICLE SIZE ANALYSIS OF SUPERFINE MATERIALS

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ABSTRACT

An evaluation was made of the Microtrac (SPA) to determine whether it would meet Atlantic Research Corporation needs for rapid analysis of superfine materials. In this size range, the Microtrac (SPA) proved to be as reproducible as the MSA particle size analyzer.

In most cases, particle size results of the Microtrac (SPA) were the same or slightly smaller than those of the MSA. However, due to shape factor, some materials tested larger by Microtrac (SPA) than by MSA.

Microtrac (SPA) analysis time is approximately six minutes for duplicate results once the sample is introduced for testing. This contrasts with MSA analysis times of 45 minutes to over 3 hours for superfine material. Good results were obtained for median particle sizes of seven micrometers down into the sub-micrometer region.

Considering that MSA particle size equipment is no longer manufactured by Mine Safety Appliances Company, the Microtrac (SPA) provides a good alternative for testing in the superfine range and has the added advantages of very rapid analysis time; simple operation; automatic accumulation, calculation and printing of data and less operator time.

INTRODUCTION

The solid propellant industry has until recently used the MSA particle size analyzer for the analysis of superfine materials. Since MSA equipment is no longer manufactured and MSA test time is fairly lengthy in the superfine region, other methods were investigated by Atlantic Research Corporation.

After investigating several instruments, it was decided that the Microtrac small particle analyzer (SPA), manufactured by the Leeds and Northrup Company, Microtrac Division, would meet the requirements of accuracy, precision, simple operation and rapid analysis time in the superfine region.

PRINCIPLE OF OPERATION (ref. 1,2)

The Microtrac (SPA) particle size analyzer (Figure 1) operates on the principle of light scattering. As the basis for analysis, the analyzer utilizes the phenomena of low-angle forward scattering and 90^{0} scattering of light in conjunction with proprietary filtering techniques.

The measurement of particle size covers the range from 0.12 micrometers to 21.1 micrometers and utilizes Fraunhofer diffraction for those particles that are significantly larger than the wavelength of the light source. As particle size approaches the wavelength of the laser source (0.63 micrometers), Mie theory must be invoked with insertion of the proper index of refraction of the sample material into the size computation. For extremely small particles (less than 0.35 micrometers in diameter), the angular distribution of scattered light flux is such that it becomes difficult to collect with a conventional optical system. Determination of particle size below 0.35 micrometers is accomplished by using 90^{0} scatter at 3 different wavelengths and 2 planes of polarization of each wavelength. Refractive index affects the relationship between forward and right-angle scatter. Compensation is carried out automatically by the system programming.

The sample to be tested is circulated through the test cell by means of a self-contained water system or by means of a peristaltic pump for organic liquids. The optical system consists of two light paths; one for the forward scatter and one for the right-angle scatter.

During part of the measurement cycle, the test cell, containing particles, is illuminated by a helium-neon laser to produce 11 channels of forward scattered light (Figure 2). The other part of the cycle sequentially places several bandpass filters and polarizers in the path of a tungsten-halogen lamp to produce 3 channels of 90^{0} scattered light (Figure 3). The two groups of histogram channels are combined and normalized together by the microprocessor program into a 14 channel histogram in the range of 0.12 micrometers to 21.1 micrometers. A typical printout of the distribution is shown in Figure 4.

Test sampling times of 8, 15, 30, 60, 120, 240, 480 or 960 seconds can be selected. A sampling time of 60 seconds provides adequate data. This produces data within 2 minutes (filter changes account for the difference in time) once the test is started. Sampling time is independent of particle size being tested.

Background contamination can be checked and automatically subtracted from the sample data by the microprocessor. Various ways of presenting the data, such as raw data, summary data, histogram data and cumulative data (% finer or % greater than diameter) may be switch selected. All data calculations are automatically performed by the microprocessor.

EVALUATION

Since the intention was to replace the MSA with the Microtrac (SPA) for particle size testing, the evaluation consisted of comparing results from the two instruments. Various materials were evaluated.

Ammonium Perchlorate

The material which gets the majority of testing is superfine ammonium perchlorate. Figure 5 shows a comparison of Microtrac (SPA) and MSA median diameters for ammonium perchlorate. A good correlation between the two instruments was found from 2.5 micrometers to 6 micrometers. Results generally were within 7% of each other with the Microtrac (SPA) results being slightly smaller than MSA results. Above 6 micrometers, a spread develops between results since some of the distribution occurs above the 21.1 micrometer limit of the Microtrac (SPA).

Figures 6 through 10 show a comparison of Microtrac (SPA) and MSA distributions for various median particle sizes of ammonium perchlorate between 2.5 micrometers and 7 micrometers. Each figure shows the log/probability distributions of the particles as detected by both instruments. For median particle sizes in the range of 2.5 to 6 micrometers, there is only a slight difference in distributions. Note that at a Microtrac (SPA) median particle size of 7 micrometers, the MSA median particle size is discernibly larger due to the fact that the Microtrac (SPA) does not see that part of the distribution above 21.1 micrometers.

One point of confusion occurs with ammonium perchlorate below one micrometer. A sample which tested as having a median diameter of 0.79 micrometers by MSA was much larger by Microtrac (SPA) analysis (1.78 micrometers). It appears that this error can be corrected by inserting the exact refractive index rather than the nominal refractive index used in the microprocessor calculations. When particle size is very small and there is not much difference between refractive indices of the material and dispersing liquid, it becomes more critical that the correct indices be used in the calculations. This correction is being investigated at this time.

In order to compare the precision of both instruments, ten individual runs of fluid energy mill (FEM) grinds were tested. Table I shows the results of those tests. The standard deviation of ten samples for the Microtrac (SPA) was approximately two-thirds that of the MSA.

TABLE I

Comparison of Microtrac (SPA) and MSA Median Particle Sizes

Run No.	Microtrac (SPA) Median Particle Size (um)	MSA Median Particle Size (um)
1	3.72, 3.68	3.90, 3.79
2	3.73, 3.68	3.95, 4.13
3	3.76, 3.69	3.90, 3.99
4	3.66, 3.69	3.94, 3.91
5	3.59, 3.60	3.74, 3.88
6	3.65, 3.63	4.01, 3.96
7	3.65, 3.65	3.79, 3.78
8	3.62, 3.62	3.85, 3.85
9	3.61, 3.57	3.74, 3.84
10	3.81, 3.80	4.03, 4.03
Mean	3.67	3.90
Standard Deviation	0.067	0.106

An interesting study which used the Microtrac (SPA) was to determine the particle size of ammonium perchlorate after it was mixed into the propellant binder. One type of propellant requires a number of fluid energy mill (FEM) runs in order to grind enough ammonium perchlorate for the mix. Microtrac (SPA) particle sizes were tested on each run and the overall average particle size calculated. The superfine ammonium perchlorate was then mixed with the propellant binder prior to addition of any other solids.

By sampling this paste and dissolving the binder, the actual particle size of the ammonium perchlorate in the mix was determined by the Microtrac (SPA). Figure 11 shows a comparison of the log/probability distribution of the calculated results versus that which was found in the paste.

Another point of interest concerned whether the Microtrac (SPA) could detect the agglomeration of ground ammonium perchlorate. A sample exposed to 50% R.H. for a period of one month showed an increase in median particle size of approximately 44%. Figure 12 indicates the increase in particle size, as detected by the Microtrac (SPA).

Heptane was used as the testing liquid and Twitchell Base 8266 as the wetting agent for Microtrac (SPA) analysis of ammonium perchlorate.

HMX (Class 5)

Class 5 HMX showed a slightly greater spread in results (Figure 13) between MSA and Microtrac (SPA) than was observed for ammonium perchlorate. The Microtrac (SPA) result is approximately 12% smaller than the MSA result. This greater spread can be explained by the fact that there are some Class 5 HMX particles

greater than the 21.1 micrometer limit of the Microtrac (SPA). For the same median size on FEM ground ammonium perchlorate, the total distribution is contained within the range of the Microtrac (SPA).

Class 5 HMX was tested on the Microtrac (SPA) using heptane as the testing liquid and lecithin (Alcolec S) as the wetting agent.

Other Materials

In addition to ammonium perchlorate and HMX, the Microtrac (SPA) has been used to test the particle size of such various materials as potassium perchlorate, ferric oxide, lead sesquioxide, zirconium carbide, aluminum, carbon black, garnet, polyvinyl chloride resin and aluminum oxide. The Microtrac (SPA) was able to detect the reduction in boron particle size as it was being ball milled (Figure 14). Heptane and Twitchell Base 8266 were also used for this test.

With some materials, when particle shape is significantly non-spherical the Microtrac (SPA) particle size is larger than that of the MSA. This is because the circulation system of the Microtrac (SPA) randomly aligns the various dimensions of each particle as the light is scattered. As a result, average diameters of the non-spherical particles are detected. Particles settling in a liquid (MSA) will align themselves in such a way as to be biased toward the smaller diameters.

Advantages

1. Once the sample is introduced into the Microtrac (SPA), duplicate results can be obtained within approximately six minutes (assuming a 60 second sampling time). This contrasts with testing times of approximately 45 minutes to over 3 hours on the MSA for superfine material. Quick analysis allows for immediate correction in the grinding process, if necessary.

2. Since the sample is continuously being circulated through the test cell, the operator can run as many replicate tests as needed to provide assurance of precise results. This, plus sampling times as short as 8 seconds, can also be used to examine different types of particle behavior such as swelling, dissolving, fracturing or agglomeration.

3. In the case of non-spherical particles, the Microtrac (SPA) will provide more of an average diameter (since it sees all dimensional configurations) whereas the MSA will be biased toward the smaller diameters.

4. The Microtrac (SPA) is very simple to operate.

5. Once the sample is introduced into the circulation system and the test started, the test proceeds without operator attention. Data is automatically accumulated, calculated and printed.

6. Both organic and inorganic materials can be tested on the Microtrac (SPA) using just about any available liquid.

7. Continuous circulation in the Microtrac (SPA) should provide a better method of keeping particles dispersed and deagglomerated than is provided, by settling, in the MSA test.

8. The Microtrac (SPA) test is independent of sample specific gravity and can accommodate blends of different densities.

9. The Microtrac (SPA) is factory-calibrated. There is no requirement for operator calibration prior to testing.

Disadvantages

1. Initial cost of the Microtrac (SPA) plus replacement costs for such items as the tungsten-halogen and laser sources.

2. Two instruments [Microtrac (Standard) and Microtrac (SPA)] are needed to cover the full range of distributions normally tested by the MSA.

CONCLUSIONS

1. Microtrac (SPA) results on most of the materials tested in the superfine region were comparable with MSA results and were as precise or more precise than MSA test data.

2. The Microtrac (SPA) has a distinct advantage of producing results much quicker than the MSA in the superfine region.

3. Other advantages include simple operation; opportunity for replicate testing; automatic accumulation, calculation and printing of data.

4. Data and conclusions in this report were developed at Atlantic Research Corporation from tests performed on the Microtrac (SPA) over a period of six months.

REFERENCES

1 Brochure on the Microtrac (SPA), Leeds and Northrup Company, Microtrac Division.
2 E. C. Muly and H. N. Frock, "Submicron Particle Size Analysis Using Light Scattering", Leeds and Northrup Company, Microtrac Division.
3 Photograph furnished by Leeds and Northrup Company, Microtrac Division.

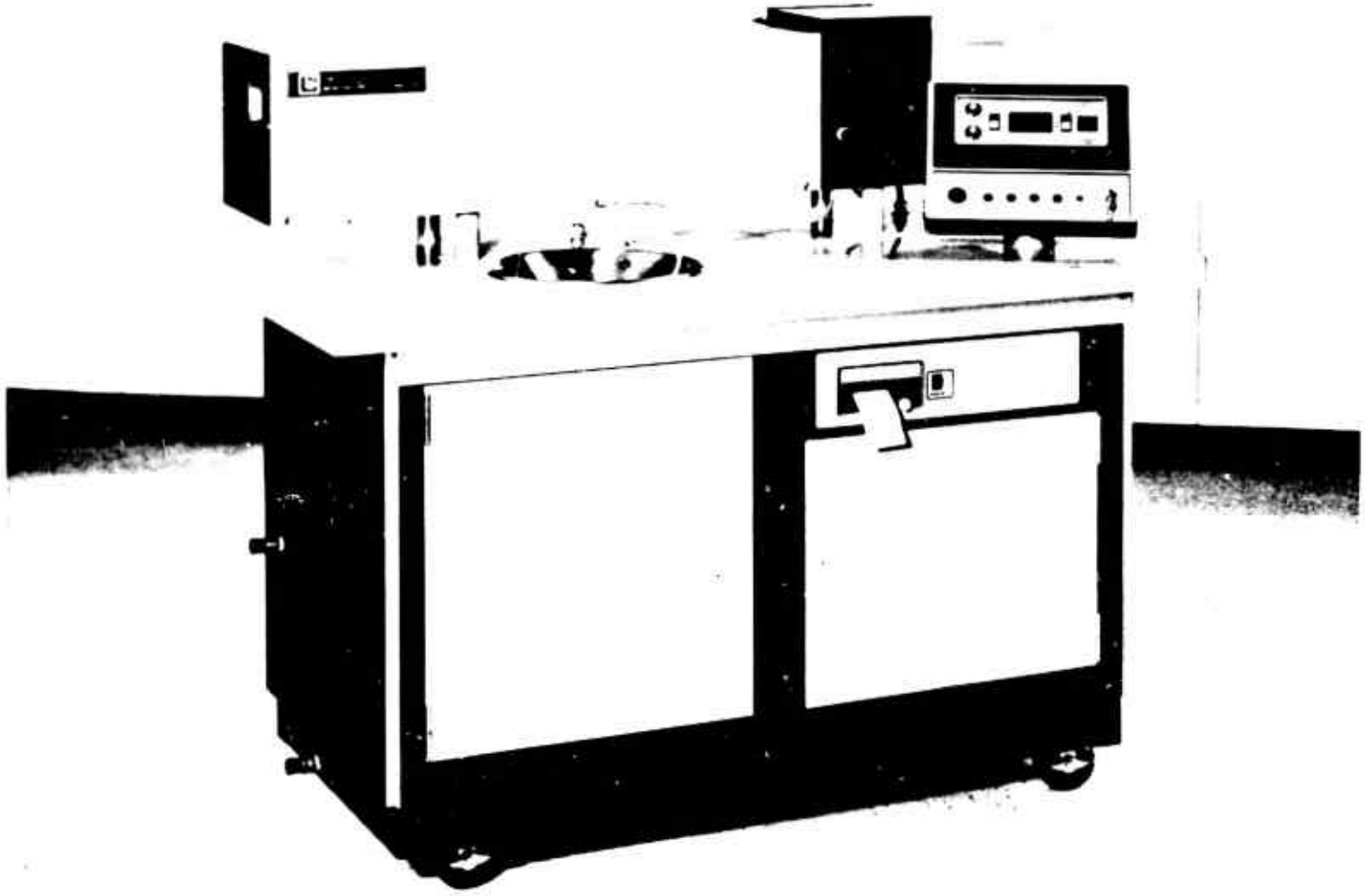

Figure 1. Microtrac Small Particle Analyzer. (Ref. 3)

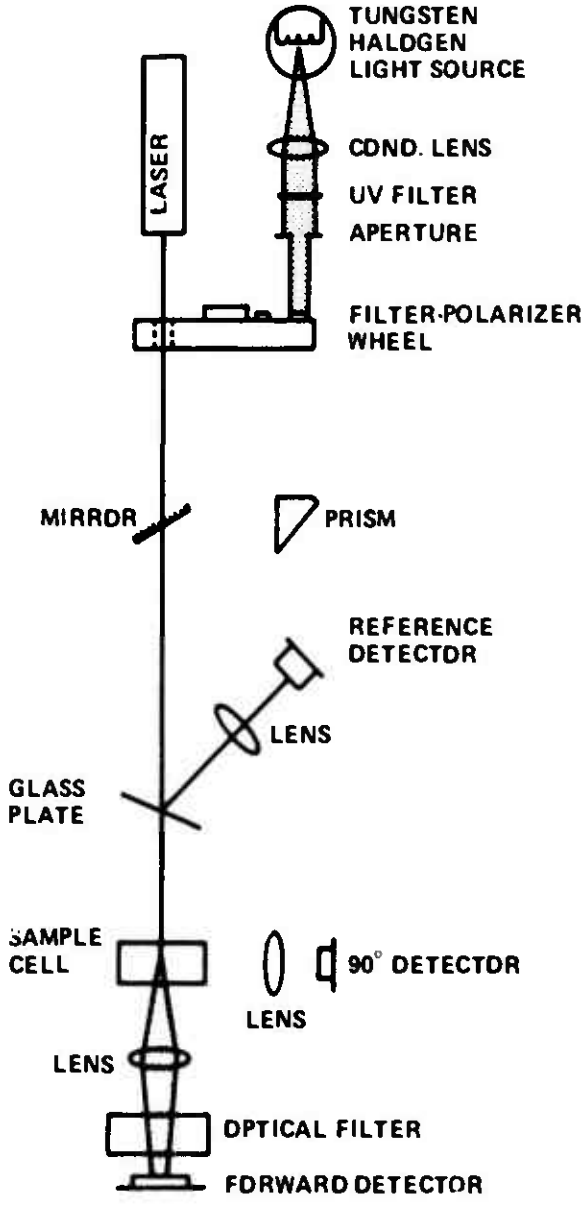

Figure 2. Forward Scattering Configuration (Ref. 2).

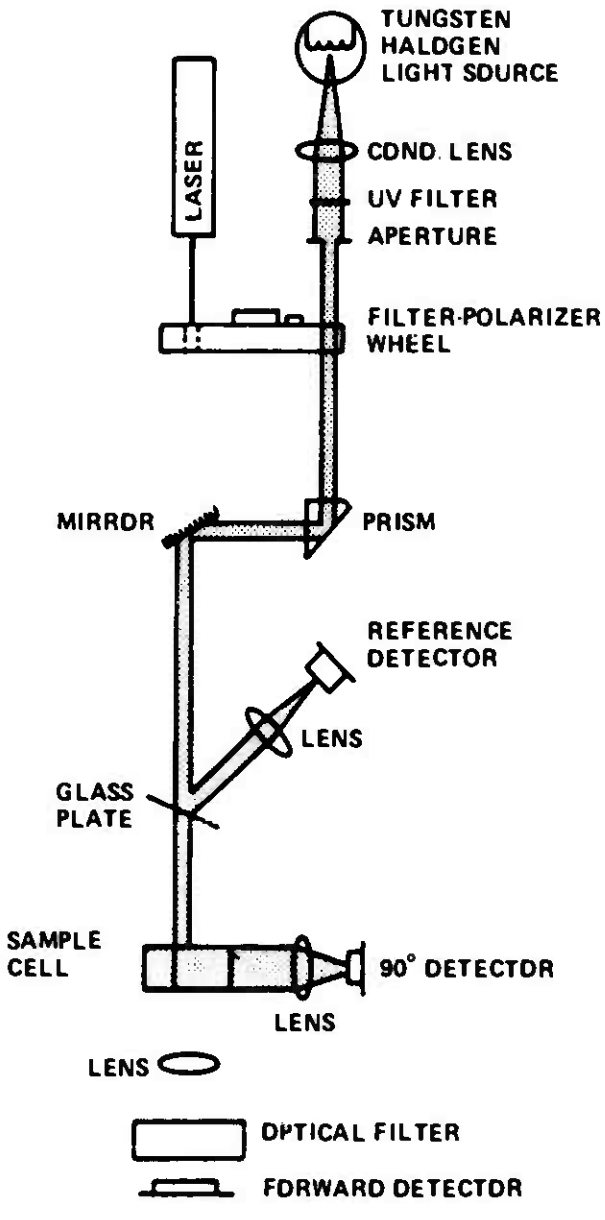

Figure 3. Side Scattering Configuration (Ref. 2).

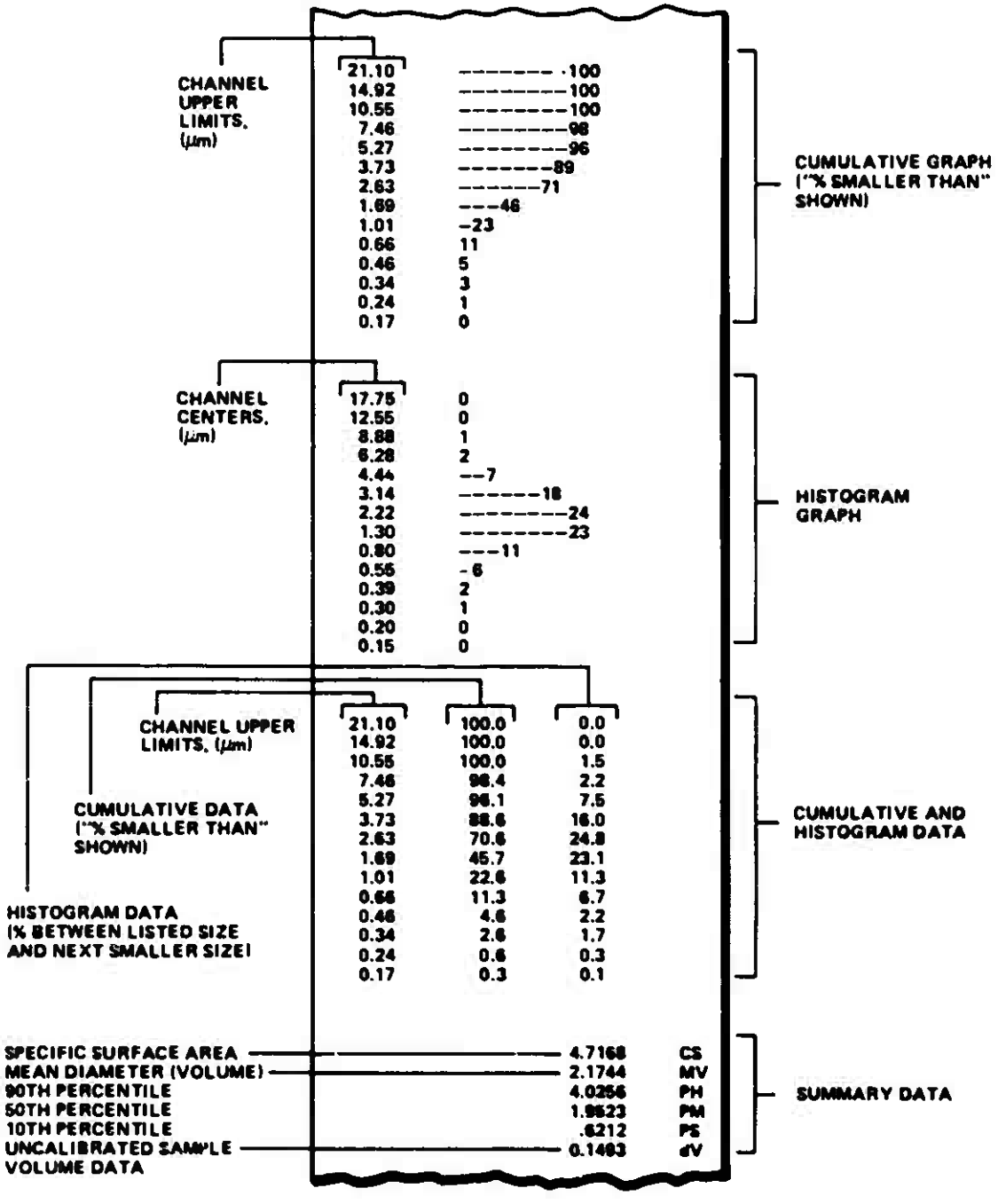

Figure 4. Typical Data Printout (Ref. 1).

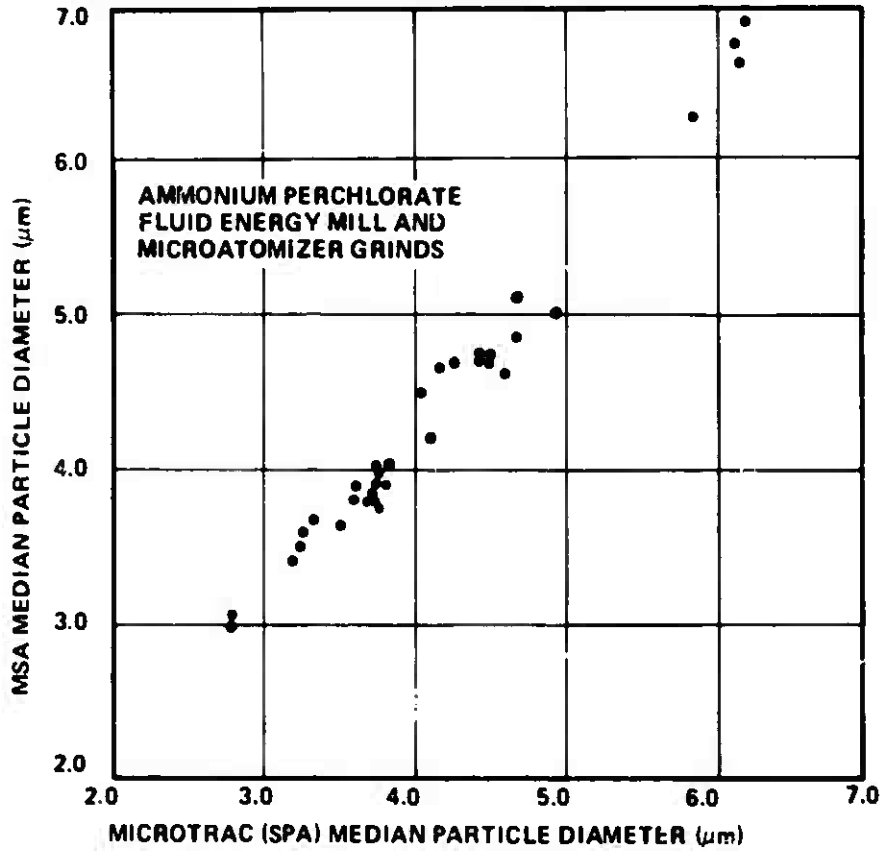

Figure 5. Comparison of Microtrac (SPA) and MSA Particle Diameters.

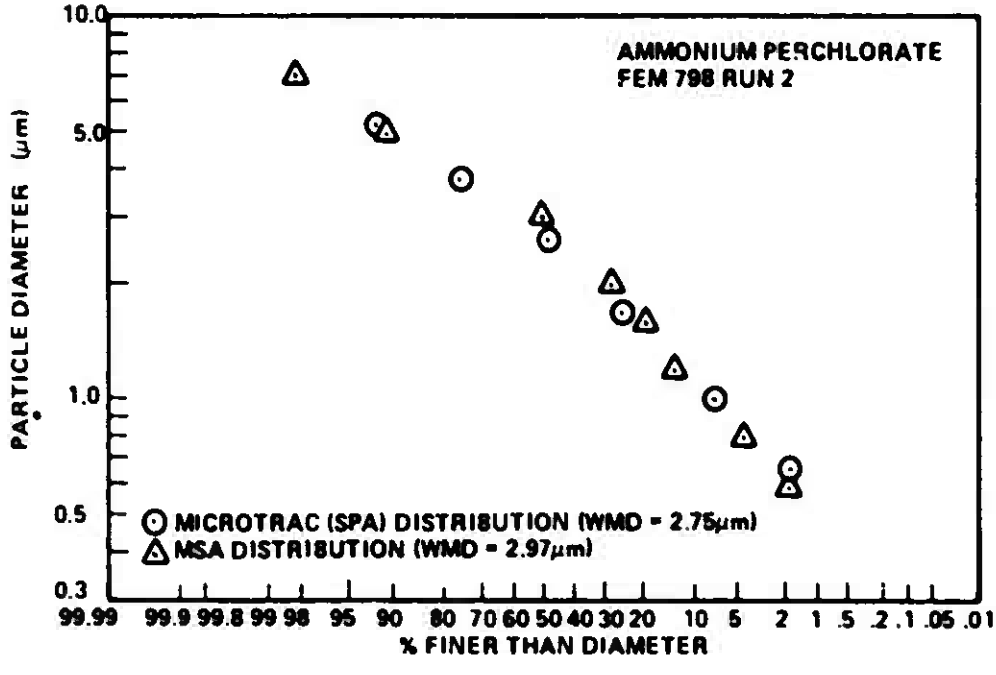

Figure 6. Comparison of Microtrac (SPA) and MSA Distributions.

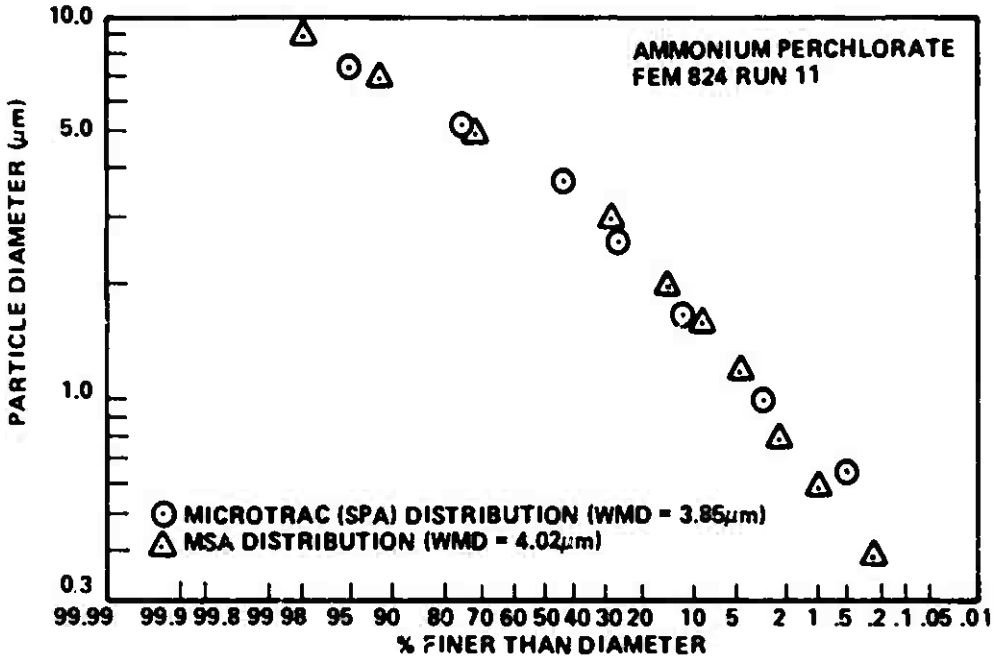

Figure 7. Comparison of Microtrac (SPA) and MSA Distributions.

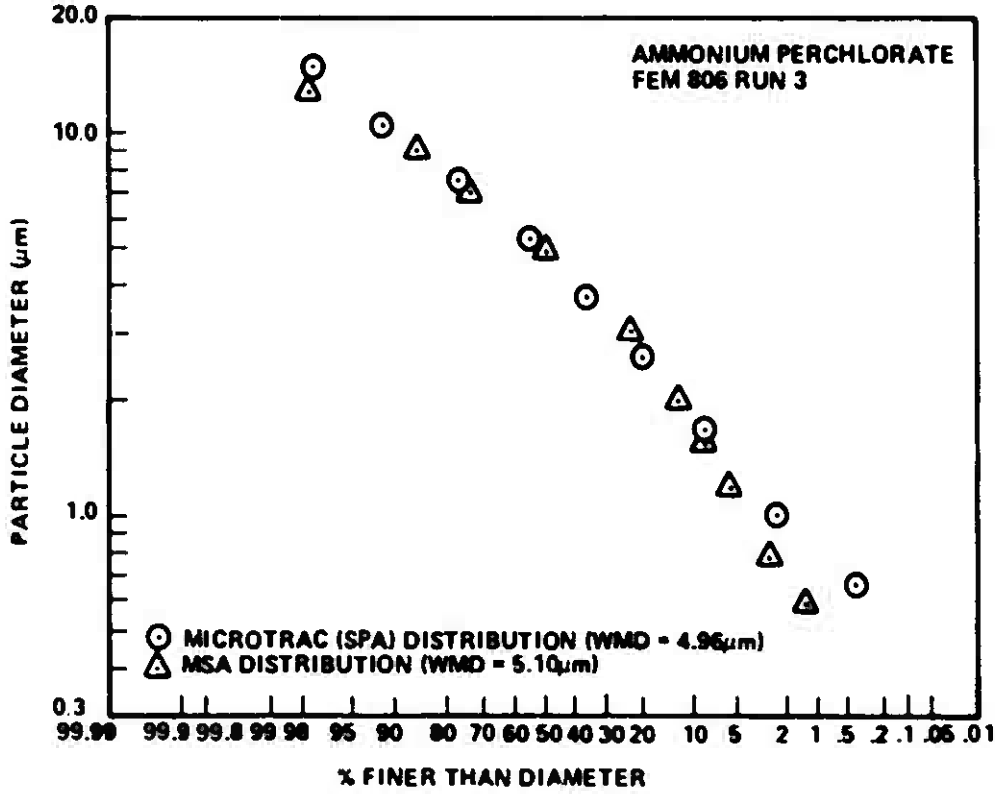

Figure 8. Comparison of Microtrac (SPA) and MSA Distributions.

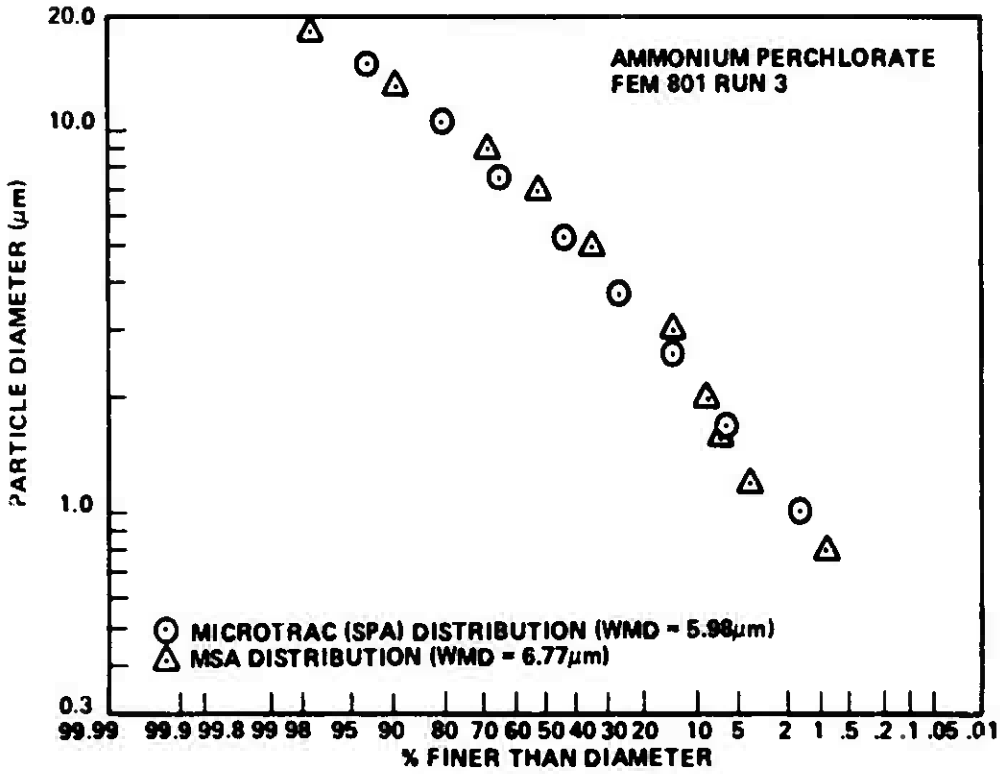

Figure 9. Comparison of Microtrac (SPA) and MSA Distributions.

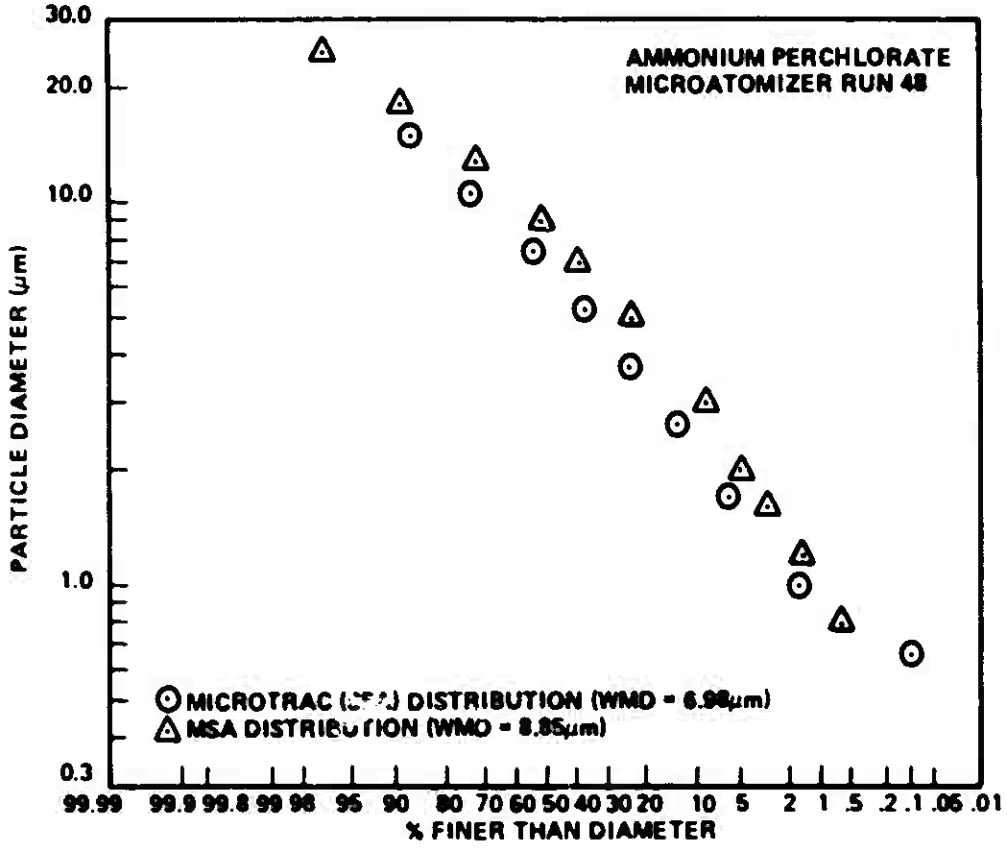

Figure 10. Comparison of Microtrac (SPA) and MSA Distributions.

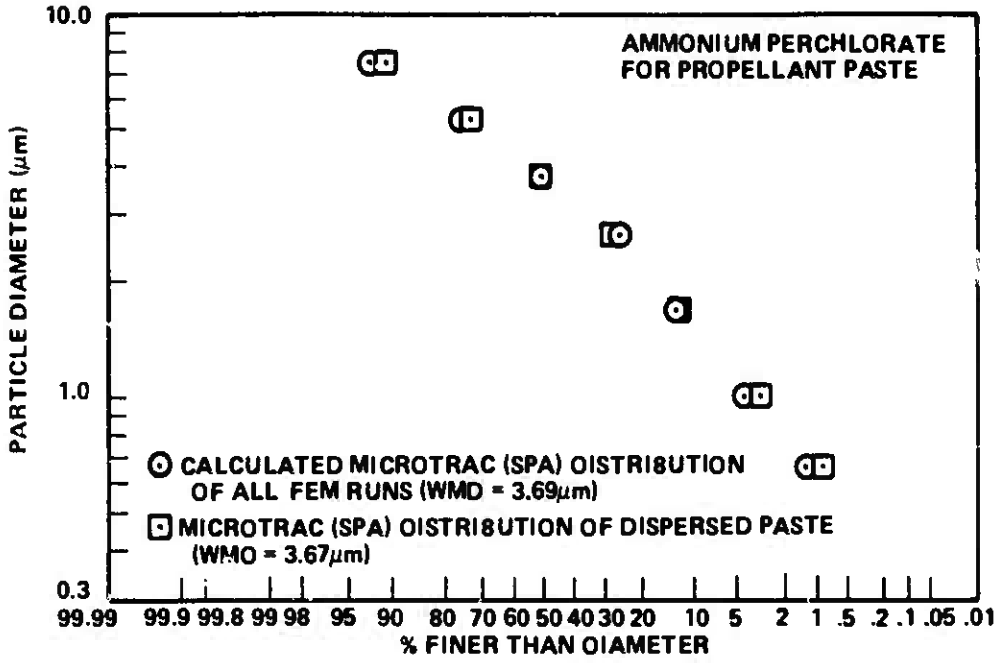

Figure 11. Calculated Avaraga Varaus Paata Diatribution by Microtrac (SPA).

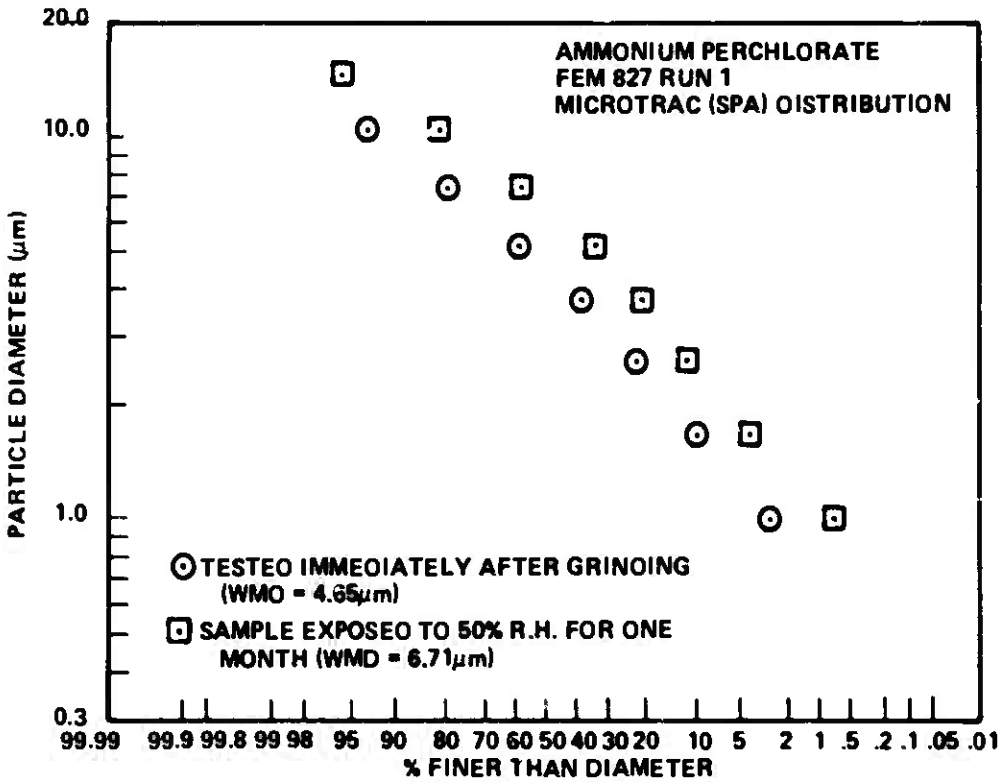

Figure 12. Change of Particle Siza Distribution Upon Sample Exposure To Humidity.

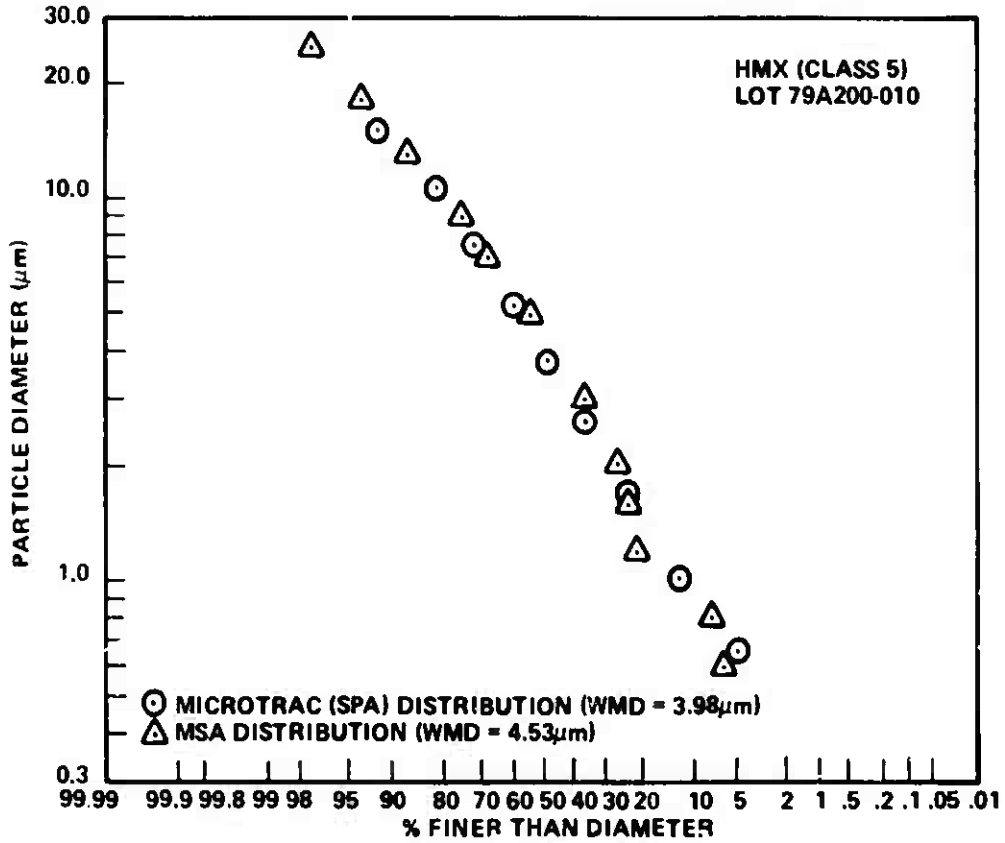

Figure 13. Comparison of Microtrac (SPA) and MSA Distributions.

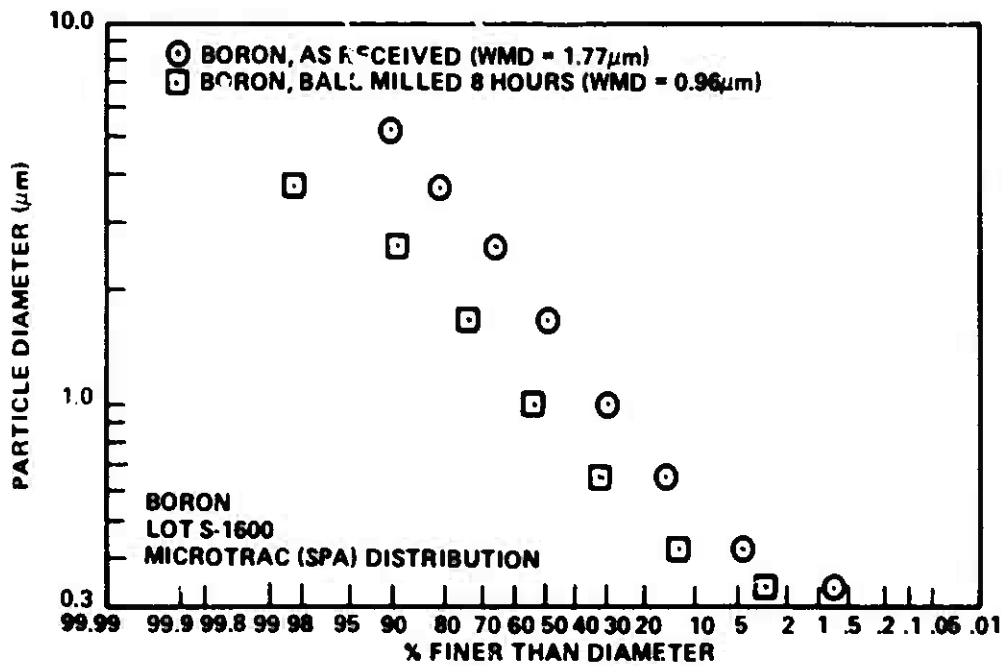

Figure 14. Change of Particle Size Distribution As a Result of Ball Milling.